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METHODS OF TEST FOR EDIBLE STARCHES AND STARCH PRODUCTS

PART I PHYSICAL METHODS

(First Revision)

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Indian Standard

METHODS OF TEST FOR EDIBLE STARCHES AND STARCH PRODUCTS

PART I PHYSICAL METHODS

(First Revision)

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AMENDMENT NO. 1 JUNE 1988

TO

IS:4706(Part 1)-1978 METHODS OF TEST FOR EDIBLE STARCHES AND STARCH PRODUCTS

PART 1 PHYSICAL METHODS

(First Revision)

(*Page 6, clause 5.3*) - Substitute the following for the existing clause:

'5.3 Take 50 ml of the paste and measure its viscosity in seconds, in the Redwood Viscometer No. 1 at 75°C.'

(AFDC 33)

Reprography Unit, BIS, New Delhi, India

Indian Standard
METHODS OF TEST FOR EDIBLE STARCHES AND STARCH PRODUCTS
PART I PHYSICAL METHODS
(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part I) (First Revision) was adopted by the Indian Standards Institution on 28 June 1978, after the draft finalized by the Edible Starches and Starch Products Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 This standard was first published in 1968 to cover comprehensively the method of test for edible starches. This revision has been taken up to up-date this standard by incorporating other methods employed for quality characteristic determination, and by expanding its scope to cover starch products as well. It is being published in two parts. This part covers physical methods; Part II of this standard covers chemical methods.

0.3 It is expected that the adoption of this standard would help in defining the quality of edible starches and starch products in a manner that would enable better quality control. Besides, it will help in achieving uniformity in the methods of test of edible starches, thereby facilitating the interpretation and comparison of results.

0.4 In the preparation of this standard, due consideration has been given to the existing methods of test followed by the edible starch industry in the country. However, wherever possible, alignment has also been made with the standards formulated by the Technical Committee ISO/TC 93 Starch (Including Derivatives and By-Products) of International Organization for Standardization.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part I) prescribes the physical methods used for testing edible starches and starch products.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. DETERMINATION OF PARTICLE SIZE

3.1 Sieves — Make a nest of two sieves, the upper being 150-micron IS Sieve (*see* IS : 460-1962†) and the lower 75-micron IS Sieve (*see* IS : 460-1962†) with a cover on top of the upper sieve and a receiver below the lower sieve.

3.2 Procedure — Weigh accurately about 100 g of the material into the upper sieve and fit it with the cover. Place the nest of sieves with the receiver in a suitable mechanical sieve shaker with tapping action and shake for 15 minutes. Stop shaking, remove the nest of sieves and transfer the residue on each sieve separately to two tared weighing dishes, using a brush, and weigh each dish.

3.3 Calculation — Calculate the percentage of the material retained on the respective sieves as follows:

a) Material retained on 75-micron IS Sieve, percent by mass $= \dfrac{100\ (M_1 + M_2)}{M}$

where

M_1 = mass in g retained on 75-micron IS sieve,

M_2 = mass in g retained on 150-micron IS sieve, and

M = mass in g taken for the test.

b) Material retained on 150-micron IS Sieve, percent by mass $= \dfrac{100\ M_2}{M}$

where

M_2 = mass in g of the material retained on 150-micron IS sieve, and

M = mass in g of the material taken for the test.

---

*Specification for water for general laboratory use (*second revision*).

†Specification for test sieves (*revised*).

4. MICROSCOPIC EXAMINATION

4.1 Apparatus

4.1.1 *Microscope* — with an eye-piece micrometer calibrated with a slide micrometer and having a magnification of 300 to 500.

4.1.2 *Microscope Slides*

4.1.3 *Cover Glasses* — either circular or square.

4.2 Procedure — Take about 1 g of the material and transfer it to a beaker containing 50 ml of water. Stir the contents of the beaker by means of a glass rod to break up granules and lumps and immediately place a drop of the suspension on a clean microscope slide using a glass tube drawn at one end to a small orifice. Press the cover glass on the drop ot the suspension taking care that no air is trapped between the cover glass and the slide. Remove the excess of liquid on the slide by means of a blotting paper. Examine the slide, so prepared, under the microscope to determine the nature of the material and compare with the photomicrograph shown in relevant Indian Standards.

5. DETERMINATION OF VISCOSITY BY REDWOOD VISCOMETER

5.1 Apparatus

5.1.1 *Redwood Viscometer No. 1*

5.1.2 *Water-Bath with a Thermo-Regulator*

5.1.3 *Time-Recording Device* — capable of reading correct to 1 second.

5.2 Procedure — From the known moisture content of the test sample calculate the amount of starch which would give 4 g of oven-dry sample. Weigh the amount exactly and transfer it to a 750-ml conical flask. Weigh the flask containing the sample on a pan balance. Add about 25 ml of cold water to the flask and make the sample into a fine slurry. Break any lumps or clots with a glass rod flattened at the end. Pour sufficient boiling water with constant stirring to avoid lump formation till the total amount of water added is just 200 g. Boil the paste gently under a reflux condenser for exactly 1 hour and cool it with constant stirring to 75°C.

5.3 Measure the viscosity of the paste in seconds in the Redwood viscometer No. 1 at 75°C.

6. EXAMINATION FOR DIRT AND OTHER SUSPENDED AND EXTRANEOUS MATTER

6.1 Procedure — Take about 10 g of the material in a 250-ml beaker and add 100 ml of water. Stir the material with a glass rod to form a suspension and allow it to stand for 2 hours. Examine the supernatant water, the surface and the bottom of the sediment for dirt or other suspended and extraneous matter.

7. DETERMINATION OF RODENT EXCRETA

7.1 Apparatus

7.1.1 *Beaker, Hooked-Lip* — 250 ml capacity.

7.1.2 *Buchner-Funnel*

7.1.3 *Ruled Paper* — 7 cm.

7.2 Reagents

7.2.1 *Chloroform*

7.2.2 *Carbon Tetrachloride*

7.3 Procedure — Weigh 50 g of the sample in 250-ml hooked-lip beaker. Add chloroform to within approximately 1 cm of top, mix thoroughly and let it settle for at least 30 minutes, stirring surface layer occasionally. Decant chloroform and the floating tissue on to the Buchner funnel, taking care not to disturb heavy residue at the bottom of the beaker. Before decanting, take care that the floating layer has not become so compact as to render this operation difficult. Add a quantity of carbon tetrachloride equal to the quantity of chloroform and tissue left in the beaker, let it settle again, and decant as before. Repeat this process with a mixture of equal parts of chloroform and carbon tetrachloride until very little tissue remains in the beaker. Wash the residue in the beaker onto a 7 cm ruled paper with a stream of chloroform or carbon tetrachloride and examine microscopically.

8. TEST FOR GELATINIZATION AND DETERMINATION OF STARCH IN GRUEL

8.1 Procedure

8.1.1 Weigh accurately about 10 g of the material in a 200-ml conical flask. Add 80 ml of boiling water into the conical flask. Connect it with an upright water-cooled condenser and heat it on a wire gauze using a bunsen burner. (It is essential that the contents of the flask begin to boil within 1 minute of adding the boiling water.) Boil the contents of the flask briskly for exactly 15 minutes, after which remove the flame, disconnect the flask from the condenser and filter the contents of the flask through 850-micron IS Sieve (*see* IS : 460-1962*). Collect the gruel in a tared porcelain dish. Plaçe the porcelain dish containing the gruel over a boiling water-bath and evaporate the water from it until the residue becomes quite dry. Remove the dish from the boiling water-bath and place it in a electric air-oven maintained at 110 ± 2°C overnight. Cool the dish in a desiccator and weigh. Repeat, heating in the oven at 110 ± 2°C, cooling and weighing at half hour intervals till the difference between two successive weighings is less than 1 mg. Note the lowest mass.

8.1.1.1 Examine the cooked globules or pearls on the sieve for their globular shape.

8.2 Calculation

Quantity of starch passed into the gruel, expressed as percentage by mass of the material taken for the test $= \frac{100\ M_1}{M}$

where

M_1 = mass in g of the dry residue obtained from the gruel, and

M = mass in g of the material taken for the test.

9. DETERMINATION OF COLOUR OF GELATINIZED ALKALINE PASTE

9.1 Apparatus

9.1.1 *Lovibond Tintometer*

9.1.2 *Porcelain Cuvette* — 1 cm, supplied by the makers with the Lovibond Tintometer.

9.2 Reagent

9.2.1 *Sodium Hydroxide Solution* — approximately 0·5 N, prepared from sodium hydroxide, analytical reagent (*see* IS : 376-1976†).

9.3 Preparation of Material — In case of coarse material take about 100 g of the material. In case of chips, finely powder in a clean mortar so that the whole of it passes through 250-micron IS Sieve (*see* IS : 460-1962*). Place this prepared material in a clean and dry stoppered glass bottle.

---

*Specification for test sieves (*revised*).

†Specification for sodium hydroxide, analytical reagent (*first revision*).

9.4 Procedure

9.4.1 Place about 10 g of the prepared material (**9.3**) in a clean and dry neutral glass beaker and add to it 95 ml of water. Heat the beaker with its contents on a boiling water-bath for about 15 minutes with continuous stirring till the material is gelatinized. Add 5 ml of sodium hydroxide solution to the gelatinized paste and stir well. Allow the slurry to cool.

9.4.2 *Examination of Gelatinized Paste* — Clean the porcelain cuvette with carbon tetrachloride to remove any oily or greasy film on it and allow it to dry. Fill the cuvette with the gelatinized paste (*see* **9.4.1**) and place it in position in the Tintometer kept in the vertical position, suitable for measuring reflected light. Place along side of it such red and/or yellow Lovibond filters as are necessary to match the shade of the gelatinized paste, observing the colour of the gelatinized paste and of the combination of Lovibond filters through the eye piece.

9.5 Report — Report the colour of the gelatinized paste in terms of Lovibond units by summing up individually the values for the red and yellow Lovibond filters as follows:

Colour reading of the gelatinized paste in the porcelain cuvette on the Lovibond Scale $= aR + by$

where

aR = the sum total of the various red Lovibond filters used, and

by = the sum total of various yellow Lovibond filters used.

10. TEST FOR GELLING POWER

10.1 Procedure

10.1.1 Weigh approximately 4 g of the material in a porcelain dish. Add 50 ml of cold water and mix. Heat to boiling over a bunsen burner, stirring the contents constantly. Boiling time shall not exceed 1 minute Remove the flame when the paste becomes transparent and begins to froth. Cool to room temperature.

10.1.1.1 The material shall be regarded to have satisfied the requirements of the test if the paste formed on cooling is too stiff to flow from the dish.

10.1.1.2 The paste formed on cooling shall be smooth, of uniform consistency and free from lumps.

11. DETERMINATION OF OUTER RESIDUAL SKIN

11.1 Procedure

11.1.1 Weigh accurately about 50 g of the material and pick out the chips with outer residual skin only. Keep them separately and weigh.

11.2 Calculation

$$\text{Material with outer residual skin, percent by mass} = \frac{100\,M_1}{M}$$

where

M_1 = mass in g of the material with outer residual skin, and

M = mass in g of the material taken for the test.

12. DETERMINATION OF COLD-WATER SOLUBLES

12.1 Procedure

12.1.1 Weigh accurately about 50 g of the test sample. Shake it with 250 ml of water at room temperature for 30 minutes. Filter the starch suspension by discarding the first 25 ml of the filtrate. Collect 100 ml of the filtrate in a dish and evaporate it to dryness on a steam-bath and dry the residue to constant weight in an air-oven at 100°C for 1 hour.

NOTE — Whatman No. 42 filter paper is suitable for this test. If the filtrate appears milky, it should be filtered again.

12.2 Calculation

$$\text{Cold-water solubles, percent by mass (on dry basis)} = \frac{M \times 2{\cdot}5 \times 10\,000}{M_1 \times (100 - X)}$$

where

M = mass in g of the residue,

M_1 = mass in g of the sample taken, and

X = moisture content percent by mass of the sample.

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